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X-Ray Studies on the Cast Structure of High Purity Aluminium in the Light of Anisotropy of the Rate of Crystal Growth. (II)

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Further to the first report, some minute examinations concerning the cast structure of the high purity aluminium melted and cast in vacuum, were performed in the light of anisotropy of the rate of crystal growth. A relation between the cast structure and the cooling velocity as well as the purity, was also studied. Further, the so-called line structure observed in the columnar crystals was examined in relation to the cooling velocity and the purity. From the back reflection X-ray analysis, it was found that the orientation difference in the line structure was from half degree to one.

I. INTRODUCTION

In the previous investigation,[1] an X-ray analysis was performed in the light of anisotropy of the rate of crystal growth, concerning the cast structure in the ingot of high purity aluminium which was melted and cast in vacuum and was produced so as to make the columnar crystals normal to the bottom of ingot. In this study, it was found that the crystals germinated at the outer round of ingot developed strongly towards the inner part of ingot, even if these crystallographic directions parallel to the direction of heat flow were far from the [001] direction (direction of easy growth). From a minute examination, it was supposed that the outer round of ingot was more rapidly cooled than the center.

Further, it was considered that the cast structure in the ingot used before did not show a typical arrangement of columnar crystals parallel to the [001] direction, probably owing to its relatively slow rate of crystal growth.

Therefore, concerning the ingot perpared under the conditions which were modified so that the melt was cooled at a more rapid cooling velocity and was cooled as uniform as possible, an X-ray study was performed from the same point of view as before. A relation between the cast structure and both the cooling velocity and the purity was also examined. Further, the so-called line structure observed in columnar crystals was examined.

II. EXPERIMENTAL PROCEDURE

1. Preparation of Specimens

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In order to prevent the outer round of ingot from being more rapidly cooled than the center, the outside of the stainless steel mould of the casting apparatus used in the previous investigation[1] was covered with a carbon steel plate and asbestos, and three gas burners were used to preheat the mould (two gas burners were used before).

The purity of aluminium used, was the same as before (99.993%). The vacuum and both the melting and the casting procedures were also similar to those in the previous study.[1]

The preparing conditions of the ingots B, H and I used for the examination are as follows:

Ingot B: The material used before was used again. The preheating temperature of the mould was 610°C and 430°C at the points P and Q in Fig. 2 in the first report[1] respectively.* The mould was cooled before preheating. The casting temperature was 800°C.

Ingot H: The material used was virgin. The casting temperature was 750°C. The mould was simultaneously cooled before casting. In order to obtain the cooling curves at both the center of ingot and its outer round, two sets of thermo-

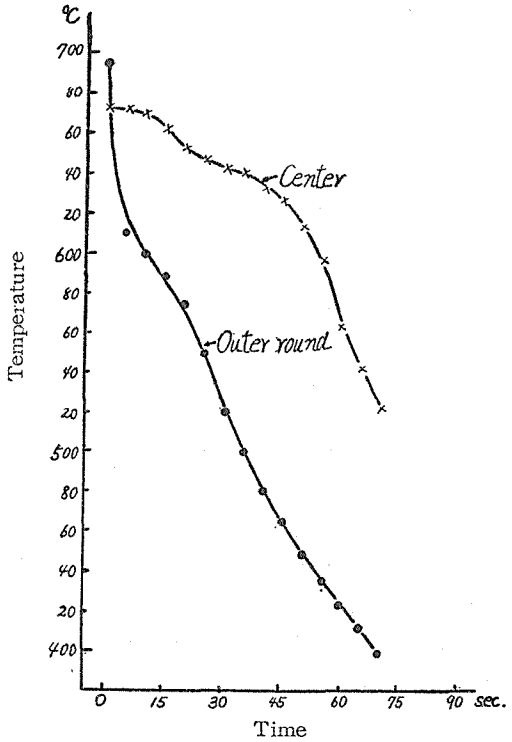

Fig. 1. Cooling curves at the center and the outer round of the ingot H.

* The points P and Q were the positions of 29 mm and 1 mm from the bottom of ingot on the center line of ingot respectively.

couple were held at the center of the mould and its inner wall at the positions of 15 mm from the bottom of the mould respectively.

Ingot I : The material used twice was used again. The casting temperature was 800°C. The mould was immediately cooled after casting. The preheating temperature of the mould was 750°C and 730°C at the points P and Q respectively.

The approximate rate of crystal growth in the ingot B obtained from the cooling curves by the same method as in Fig. 2 in the first report,[1] was about 168 mm per minute. The cooling curves of the ingot H are given Fig. 1.

2. Experimental Method

In order to perform an X-ray analysis, the plate-form specimens J and K were cut off from the half of the ingot H vertically divided equally at its center, in the same manner as in Fig. 4 in the first report,[1] The macrostructures of these specimens were revealed by etching in the mixed solution used by Chalmers et al.[2] after polishing with emery papers.* The plate-from specimen L was also cut off from the half of the ingot B in the same manner as in the specimen J mentioned above and the line structure was revealed by etching in hydrochloric acid. Further, the plate-from specimen M cut off from the ingot I by the same method as in the specimens J and L, was etched in the mixed solution mentioned above.

The same method as in the first report[1] was adopted for the X-ray analysis with respect to columnar crystals. As for the line structure observed in columnar crystals, the back reflection Laue method was applied.

III. EXPERIMENTAL RESULTS AND DISCUSSIONS

1. Macrostructure

The macrostructures of the specimens J and K are shown in Figs. 2 and 3, and those of the specimens L and M also in Figs. 4 and 5. Crystals observed in

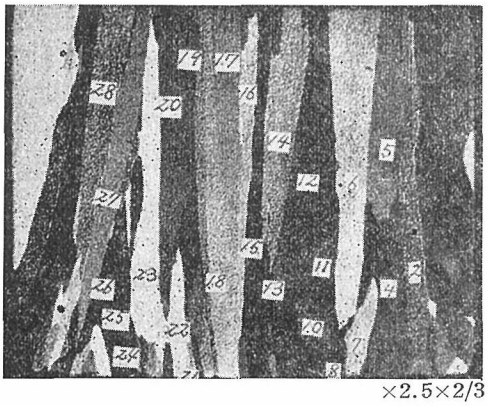

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Fig. 2. Macrostructure of the specimen J.

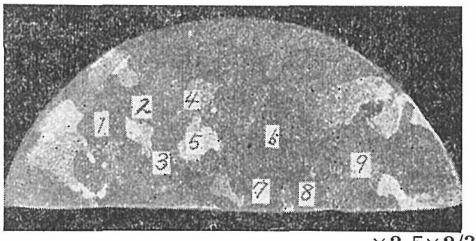

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Fig. 3. Macrostructure of the specimen K.

* The proportion of this mixed solution is 9 parts hydrochloric acid, 3 parts nitric acid, 2 parts hydrofluoric acid and 5 parts water.

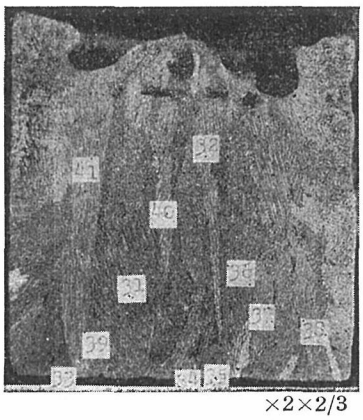

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Fig. 4. Macrostructure of the specimen L.

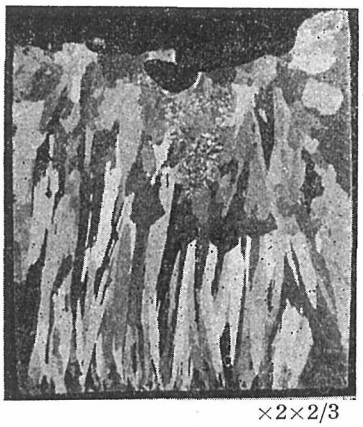

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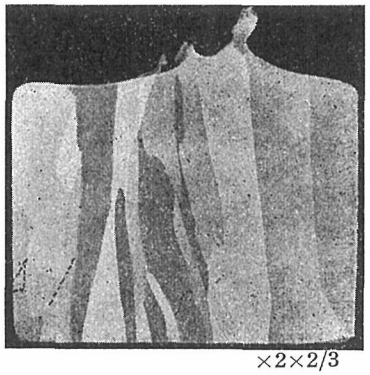
Fig. 5. Macrostructure of the specimen M



×2×2/3

Fig. 6. Macrostructure of the specimen C in the first report.

Figs. 2, 4 and 5 are smaller in size than those in Fig. 6 (Fig. 5 in the first report[1] was reproduced here). The above mentioned relation is attributable to the fact that the approximate rate of crystal growth of the specimnn in Fig. 4 is about 168 mm per minute and that of the specimen in Fig. 6 is about 67 mm per minute: i. e. the faster the cooling velocity of the specimen, the smaller becomes the size of columnar crystals in the specimen, and this result coincides with that obtained by Kondic et al.[3]

In the cast structure of pure metals, many small chill crystals germinate generally at the layer of the melt close to the cold metal mould. However, in the case of high purity aluminium, few chill crystals are observed as shown in Fig. 2, 4 and 6.* The phenomenon mentioned above has also been observed by Kondic et al.[3] and it may be because aluminium is easy to supercool but difficult to

* Since the material had already been used twice, the purty of the specimen in Fig. 5 lowered and chill crystals germinated at the bottom.

germinate. Crystals in the specimen in Fig. 5 are much smaller than those in Fig. 6, and the so-called equiaxed crystals are observed at the inner part in the former, despite the fact that both specimens were cast almost under the same conditions. This phenomenon is attributable to the lowering of purity.

2. Results of X-Ray Analysis

It was found from Fig. 1 that the outer round of ingot was rapidly cooled as had been the case with the first report,[1] in spite of the various modifications. Therefore, the crystals that developed at the outer round of ingot, were omitted from the X-ray analysis.

The stereographic projection points of the crystals observed in Fig. 2 whose directions are normal to the bottom of ingot, are given in Fig. 7. The numbers given to the crystals have no relation with those in the first report.[1] The crystals not denoted with numbers were omitted from the analysis, on account of the superpositions with another crystals developed behind the surface.

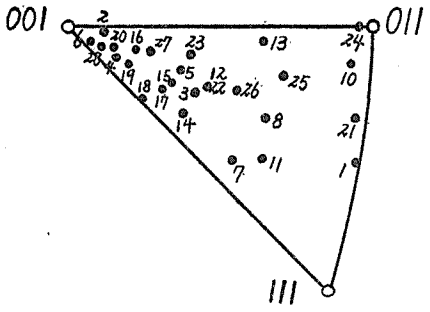


Fig. 7. Stereographic projection points of the crystals in Fig. 2 whose directions are perpendicular to the bottom of ingot.

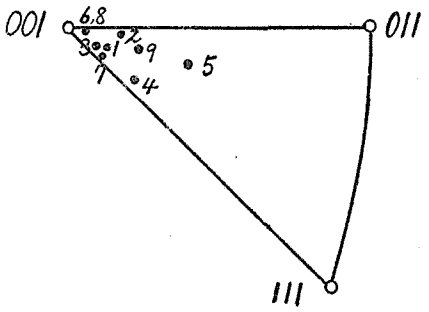


Fig. 8. Stereographic projection points of the crystals in Fig 3 whose directions are perpendicular to the bottom of ingot.

The crystals 2, 6, 16, 20, 27 and 28, whose directions normal to the bottom of ingot are oriented nearly in the [001] direction, develop strongly. The crystals 15, 17and 18 oriented relatively near the [001] direction, grow also strongly. The crystal 14 is shorter, despite its situation near the [001] direction, but is longer behind the surface, while the crystals 1, 7, 8, 10, 11, 13, 21, 24, 25 and 26 which are situated far from the [001] direction, disappear midway.

As for nine relatively large crystals among the crystals existing in the residual part of the specimen in Fig. 3, except for the outer round, the stereographic projection points whose directions are normal to the bottom of ingot, are given in Fig. 8:* i. e. all the crystals except for 5, are generally near the [001] direction.

From the result of the X-ray analysis mentioned above concerning the specimens J and K, it may be maintained that the crystals whose [001] directions are

* The numbers denoted for the specimen shown in Fig. 3 are not related to those in Fig. 2.

near the direction of heat flow, develop strongly at the residual portion except the outer round of ingot.

3. Line Structure in Columnar Crystals

The lines observed in the columnar crystals in the specimen in Fig. 4 correspond to one of the line structures observed in tin single crystals which are being investigated by the authors[4] (called "corrugation" structure by Chalmers[5]). The directions of lines varied with columnar crystals. In the specimens of higher purity shown in Fig. 6 than that in Fig. 4, the line structure is difficult to appear by etching in hydrochloric acid and the spacing between the lines is wider than that in Fig. 4, owing to its slower rate of crystal growth than in Fig. 4.*

From the back reflection Laue analysis, it was found that the orientation differences in the line structure were from half degree to one, an example of which is given in Fig. 9.

The stereographic projection points of the columnar crystals 31~41 in Fig. 4 are given in Fig. 10 in which the center is the direction normal to the bottom of ingot. In this case these projection points are those of the [001] direction nearest to the direction normal to the bottom of ingot. In the crystals whose [001] directions are near the center of figure (e. g. the crystals 32, 38 and 41), the line structures near the [001] direction are observed, whereas those observed in the crystals far from the center are complex.**

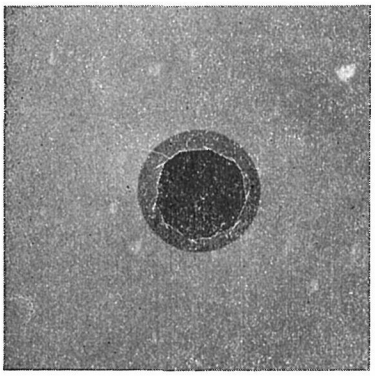


Fig. 9. X-ray back reflection pattern.

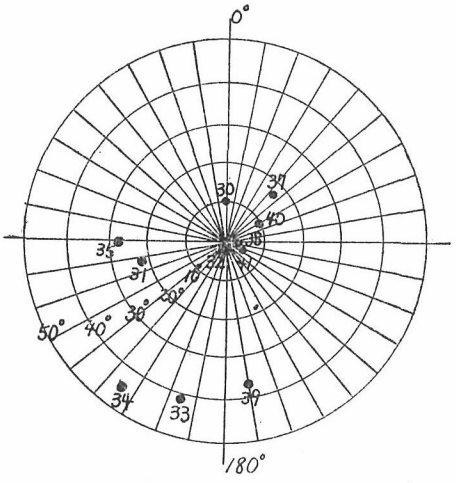


Fig. 10. Stereographic projection points of the crystals in Fig. 4, whose directions are parallel to [001] which is nearest to the center of the figure. The center is normal to the bottom of ingot.

* It seems that the spacing between the lines was strongly effected by the cooling velocity than by the impurity.

** As for the stainless steel welded, it has been reported by Prince[6] that the line structures are observed in columnar crystals of the welded part but the three dimentional cell structures are observed in equlaxed crystals.

4. Dendrite Structure

It was mentioned in the first report[1] that the dendrite structure was observed at the side surface of ingot. This dendrite structure is observed from the surface to the part of a distance, at the upper half of crystals developed at the outer round of ingot. Its example is given in Fig. 11, showing that the dendrite structures

×15

Fig. 11. Dendrite structure on the surface of a certain crystal.

developed in the mutually opposite direction in the midway of growth at the surface of a certain crystal. This figure also implies the complex heat flow at the ingot surface.

Further, it was found that the direction of dendrite did not always coincide with the special crystallographic direction (e. g. the [001] direction in cubic metals). The problem of the dendrite structure will be discussed in the study on the line structures in tin single crystals.[4]

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